PURGEABLE ORGANICS BY PURGE AND TRAP GC/MS
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Facility Name:_____VELAP ID_____

Assessor Name:_____Analyst Name:_____Inspection Date_____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst:_____ Sample ID: _____ Date of Sample Preparation:_____ Date of Analysis:_____					
1. Are sample vials 25 mL or larger, glass, equipped with screw cap with a hole, detergent washed, rinsed with tap and DI water, and dried at 105°C before use?	5.1.1, 9.2				
2. Are septa Teflon-faced silicon, washed as above, and dried for 1 hour at 105°C?	5.1.2				
3. Can purge device accept 5 mL of sample with a water column at 3 cm deep?	5.2.1				
4. Is trap at least 25 cm long with at least 0.105 inch inside diameter? Are adsorbents packed using minimum lengths of 1 cm methyl silicone, 15 cm Tenax (2,6-diphenylene oxide polymer), and 8 cm silica gel?	5.2.2				
5. Is the desorber capable of rapidly heating the trap to 180°C?	5.2.3				
6. Is the GC column 6 ft x 0.1 inch inner diameter, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent?	5.3.2				
7. If residual chlorine is present, is sodium thiosulfate added to the empty bottle prior to sampling?	9.1				
8. Are samples iced or refrigerated from time of collection until analysis?	9.1				
9. Are sample vials sealed with no entrapped air bubbles?	9.2				
10. For benzene, toluene, and ethyl benzene, is a second sample collected and preserved at a pH of about 2 if samples will not be analyzed within 7 days?	9.3				
11. Are all sample analyzed within 14 days of collection?	9.4				
12. Are stock standards stored with minimal headspace using Teflon lined caps at -10 to -20°C in the dark?	6.5.4				
Notes/Comments:					

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
13. Are standards prepared weekly for bromomethane, chloroethane, chloromethane, vinyl chloride, and 2-Chloro- ethylvinyl ether and at least monthly for all other standards?	6.5.5				
14. Is the trap conditioned 10 minutes once daily prior to use?	7.1				
15. For internal standard calibration, is the calibration a minimum of 3 concentration levels for each parameter, with one near but above the MDL?	7.3.1				
16. Is the working calibration curve verified on each working day by analyzing a QC check sample which contains 20 µg/L of each parameter? Acceptance criteria in Table 5 must be met, or the test repeated for failed parameters.	7.4, 8.2.2				
17. Is demonstration of capability performed using a standard containing 20 µg/L of each parameter in methanol?	8.2				
18. Are at least 5% of samples from each sample site spiked at the regulatory limit or 1–5 times background concentration, whichever is higher; if no regulatory limit, are they spiked at 20 µg/L or 1-5 times background concentration, whichever is higher? (Minimum of one spike per month.)	8.1.4, 8.3				
19. Do spike recoveries meet the criteria in Table 5 or the optional criteria using Table 6? If criteria are not met, is a QC check standard containing 20 µg/L of each failed parameter analyzed and assessed per Table 5?	8.3 - 8.4				
20. Are all samples spiked with three surrogate compounds from Table 3, and is the percent recovery calculated?	8.5, 6.7				
21. Is purge gas (helium) flow rate adjusted to 40 mL/min?	11.3				
22. Is 5.0 mL of sample injected?	11.4				
23. Is sample purged for 11.0 minutes ± 6 seconds?	11.6				
24. Is trap desorbed at 180°C while backflushing the trap with an inert gas at 20-60 mL/min. for four minutes?	11.7				

Notes/Comments:

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
25. After desorbing, is the trap reconditioned by returning to purge mode at 180°C, waiting seven minutes, and allowing the trap to cool between samples?	11.9				
26. For qualitative identification, did the characteristic masses of each parameter maximize in the same or within one scan of each other?	12.1.1				
27. For qualitative identification, are retention times for identified parameters within ±30 seconds of the retention time of the authentic compounds?	12.1.2				
28. For qualitative identification, did the relative peak heights of the three characteristic masses in the Extracted Ion Current Profile agree within 20% of the relative intensities of these masses in a reference mass spectrum?	12.1.3				
Notes/Comments:					

Table 3—Suggested Surrogate and Internal Standards

Compound	Retention time (min)[a]	Primary m/z	Secondary masses
Benzene d-6	17.0	84	
4-Bromofluorobenzene	28.3	95	174, 176
1,2-Dichloroethane d-4	12.1	102	
1,4-Difluorobenzene	19.6	114	63, 88
Ethylbenzene d-5	26.4	111	
Ethylbenzene d-10	26.4	98	
Fluorobenzene	18.4	96	70
Pentafluorobenzene	23.5	168	
Bromochloromethane	9.3	128	49, 130, 51
2-Bromo-1-chloropropane	19.2	77	79, 156
1,4-Dichlorobutane	25.8	55	90, 92

[a]For chromatographic conditions, see Table 1.

Table 5—Calibration and QC Acceptance Criteria-Method 624[a]

Parameter	Range for Q (µ/g/L)	Limit for s (µ/g/L)	Range for $\bar{X}$ (µ/g/L)	Range for P, P_s (%)
Benzene	12.8 - 27.2	6.9	15.2 - 26.0	37 - 151
Bromodichloromethane	13.1 - 26.9	6.4	10.1 - 28.0	35 - 155
Bromoform	14.2 - 25.8	5.4	11.4 - 31.1	45 - 169
Bromomethane	2.8 - 37.2	17.9	D - 41.2	D - 242
Carbon tetrachloride	14.6 - 25.4	5.2	17.2 - 23.5	70 - 140
Chlorobenzene	13.2 - 26.8	6.3	16.4 - 27.4	37 - 160
Chloroethane	7.6 - 32.4	11.4	8.4 - 40.4	14 - 230
2-Chloroethylvinyl ether	D - 44.8	25.9	D - 50.4	D - 305
Chloroform	13.5 - 26.5	6.1	13.7 - 24.2	51 - 138
Chloromethane	D - 40.8	19.8	D - 45.9	D - 273
Dibromochloromethane	13.5 - 26.5	6.1	13.8 - 26.6	53 - 149
1,2-Dichlorobenzene	12.6 - 27.4	7.1	11.8 - 34.7	18 - 190
1,3-Dichlorobenzene	14.6 - 25.4	5.5	17.0 - 28.8	59 - 156
1,4-Dichlorobenzene	12.6 - 27.4	7.1	11.8 - 34.7	18 - 190
1,1-Dichloroethane	14.5 - 25.5	5.1	14.2 - 28.5	59 - 155
1,2-Dichloroethane	13.6 - 26.4	6.0	14.3 - 27.4	49 - 155
1,1-Dichlorothene	10.1 - 29.9	9.1	3.7 - 42.3	D - 234
trans-1,2-Dichloroethene	13.9 - 26.1	5.7	13.6 - 28.5	54 - 156
1,2-Dichloropropane	6.8 - 33.2	13.8	3.8 - 36.2	D - 210
cis-1,3-Dichloropropene	4.8 - 35.2	15.8	1.0 - 39.0	D - 227
trans-1,3-Dichloropropene	10.0 - 30.0	10.4	7.6 - 32.4	17 - 183
Ethyl benzene	11.8 - 28.2	7.5	17.4 - 26.7	37 - 162
Methylene chloride	12.1 - 27.9	7.4	D - 41.0	D - 221
1,1,2,2-Tetrachloroethane	12.1 - 27.9	7.4	13.5 - 27.2	46 - 157
Tetrachloroethene	14.7 - 25.3	5.0	17.0 - 26.6	64 - 148
Toluene	14.9 - 25.1	4.8	16.6 - 26.7	47 - 150
1,1,1-Trichloroethane	15.0 - 25.0	4.6	13.7 - 30.1	52 - 162
1,1,2-Trichloroethane	14.2 - 25.8	5.5	14.3 - 27.1	52 - 150
Trichloroethene	13.3 - 26.7	6.6	18.6 - 27.6	71 - 157
Trichlorofluoromethane	9.6 - 30.4	10.0	8.9 - 31.5	17 - 181
Vinyl chloride	0.8 - 39.2	20.0	D - 43.5	D - 251

Q = Concentration measured in QC check sample, in µg/L (Section 7.5.3).
s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$ = Average recovery of four recovery measurements, in µg/L (Section 8.2.4).
P, P_s = Percent recovery measured, (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.
[a]Criteria were calculated assuming a QC check sample concentration of 20 µg/L.

NOTE: These criteria are based directly upon the method performance data in Table 6. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 6.

Table 6—Method Accuracy and Precision as Functions of Concentration—Method 624

Parameter	Accuracy, as recovery, X′ (µ/g/L)	Single analyst precision, s_r' (µ/g/L)	Overall precision, S′ (µ/g/L)
Benzene	0.93C+2.00	0.26$\bar{X}$-1.74	0.25$\bar{X}$-1.33
Bromodichloromethane	1.03C-1.58	0.15$\bar{X}$+0.59	0.20$\bar{X}$+1.13
Bromoform	1.18C-2.35	0.12$\bar{X}$+0.36	0.17$\bar{X}$+1.38
Bromomethane[a]	1.00C	0.43$\bar{X}$	0.58$\bar{X}$
Carbon tetrachloride	1.10C-1.68	0.12$\bar{X}$+0.25	0.11$\bar{X}$+0.37
Chlorobenzene	0.98C+2.28	0.16$\bar{X}$-0.09	0.26$\bar{X}$-1.92
Chloroethane	1.18C+0.81	0.14$\bar{X}$+2.78	0.29$\bar{X}$+1.75
2-Chloroethylvinyl ether[a]	1.00C	0.62$\bar{X}$	0.84$\bar{X}$
Chloroform	0.93C+0.33	0.16$\bar{X}$+0.22	0.18$\bar{X}$+0.16
Chloromethane	1.03C+0.81	0.37$\bar{X}$+2.14	0.58$\bar{X}$+0.43
Dibromochloromethane	1.01C-0.03	0.17$\bar{X}$-0.18	0.17$\bar{X}$+0.49
1,2-Dichlorobenzene[b]	0.94C+4.47	0.22$\bar{X}$-1.45	0.30$\bar{X}$-1.20
1,3-Dichlorobenzene	1.06C+1.68	0.14$\bar{X}$-0.48	0.18$\bar{X}$-0.82
1,4-Dichlorobenzene[b]	0.94C+4.47	0.22$\bar{X}$-1.45	0.30$\bar{X}$-1.20
1,1-Dichloroethane	1.05C+0.36	0.13$\bar{X}$-0.05	0.16$\bar{X}$+0.47
1,2-Dichloroethane	1.02C+0.45	0.17$\bar{X}$-0.32	0.21$\bar{X}$-0.38
1,1-Dichloroethene	1.12C+0.61	0.17$\bar{X}$+1.06	0.43$\bar{X}$-0.22
trans-1,2,-Dichloroethene	1.05C+0.03	0.14$\bar{X}$+0.09	0.19$\bar{X}$+0.17
1,2-Dichloropropane[a]	1.00C	0.33$\bar{X}$	0.45$\bar{X}$
cis-1,3-Dichloropropene[a]	1.00C	0.38$\bar{X}$	0.52$\bar{X}$
trans-1,3-Dichloropropene[a]	1.00C	0.25$\bar{X}$	0.34$\bar{X}$
Ethyl benzene	0.98C+2.48	0.14$\bar{X}$+1.00	0.26$\bar{X}$-1.72
Methylene chloride	0.87C+1.88	0.15$\bar{X}$+1.07	0.32$\bar{X}$+4.00
1,1,2,2-Tetrachloroethane	0.93C+1.76	0.16$\bar{X}$+0.69	0.20$\bar{X}$+0.41
Tetrachloroethene	1.06C+0.60	0.13$\bar{X}$-0.18	0.16$\bar{X}$-0.45
Toluene	0.98C+2.03	0.15$\bar{X}$-0.71	0.22$\bar{X}$-1.71
1,1,1-Trichloroethane	1.06C+0.73	0.12$\bar{X}$-0.15	0.21$\bar{X}$-0.39
1,1,2-Trichloroethane	0.95C+1.71	0.14$\bar{X}$+0.02	0.18$\bar{X}$+0.00
Trichloroethene	1.04C+2.27	0.13$\bar{X}$+0.36	0.12$\bar{X}$+0.59
Trichloroflouromethane	0.99C+0.39	0.33$\bar{X}$-1.48	0.34$\bar{X}$-0.39
Vinyl chloride	1.00C	0.48$\bar{X}$	0.65$\bar{X}$

X′ = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.
S_r = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
S′ = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
C = True value for the concentration, in µg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in µg/L.
[a]Estimates based upon the performance in a single laboratory[13].
[b]Due to chromatographic resolution problems, performance statements for these isomers are based upon the sums of their concentrations.